Soluble Chlorine in Animal Feed

Scope

The results from this method are reported as % NaCl. Since this method determines the total soluble chloride in animal feed, samples containing chloride sources other than salt (sodium chloride) should not be analyzed by this method and reported as % NaCl.

Summary

A ground portion of sample is mixed with water to dissolve the chloride. The dissolved chloride is potentiometrically titrated with 0.1N $AgNO_3$.

Comments

Ingredient labels should be checked to find out if the product contains chloride sources other than salt.

Apparatus and Materials

 A. 400 ml beakers.

 B. 200 ml volumetric flask.

 C. Potentiometer or titrator.

 D. Ag-AgCl reference electrode.

 E. Ag-indicating electrode.

Reagents

 A. 0.1N $AgNO_3$: 17.0 g $AgNO_3$/liter deionized water.

 B. Concentrated HNO_3.

C.   High purity NaCl.

Procedure

A.   Standardization of 0.1N $AgNO_3$ solution:

1.   Dry high purity NaCl for 2 hours at 105°.

2.   Weigh about 125 mg dry NaCl into a 400 ml beaker. Record the weight to the nearest 0.0001 g.

3.   Add 200 ml deionized H_2O and 1 ml HNO_3.

4.   Null potentiometer and titrate NaCl solution with 0.1N $AgNO_3$ solution. Add titrant in small enough increments so that the voltage end point is obvious.

5.   Plot ml $AgNO_3$ solution against mv or scale readings.

6.   The voltage end point is the inflection point of the titration curve.

7.   To determine the inflection point, draw 2 straight lines having a 45° slope with respect to the titration curve axes and tangent to the curve at the 2 points of greatest curvature. Construct a line parallel to and midway between these two lines. The inflection point is the intersection of this line and the titration curve.

8.   Repeat the standardization process at least twice more, titrating to the voltage end point determined from the first standardization. Record the ml of 0.1N $AgNO_3$ required for each titration.

9.   Calculate the grams of NaCl that each ml of $AgNO_3$ titrates from each standardization run by dividing the weight of NaCl (in grams) by the ml of $AgNO_3$ required to titrate to the end point. Calculate the average of the results from the three standardizations.

B.   Analysis of samples containing less than 5% sodium chloride:

1.   Weigh about 5.8 grams of sample into a 400 ml beaker and record the weight to the nearest 0.0001 g.

2.   Add about 200 ml deionized H_2O and 1 ml HNO_3.

3.   Swirl the mixture gently and let stand 10 minutes for complete solution of chlorides.

4.   Titrate with 0.1N $AgNO_3$ to the same voltage end point as determined from the standardization.

5.   Record the ml of $AgNO_3$ required for the titration.

C.   Analysis of samples containing more than 5% sodium chloride:

1.   Weigh about 5.8 g of sample and transfer to a 200 ml volumetric flask. Record the weight to the nearest 0.0001 g.

2.   Add about 190 ml deionized H_2O and 1 ml HNO_3.

3.   Dilute to volume with deionized H_2O, mix, and let stand 10 minutes.

4.   Transfer an aliquot containing about 125 mg NaCl to a 400 ml beaker. To determine the appropriate sized aliquot, divide the sample weight (g) by the dilution volume (ml) and multiply by 1000 to convert to milligrams. Divide 125 milligrams by the milligrams/ml obtained from this calculation to find the aliquot size (ml) to use for titration.

5.   Dilute to about 200 ml with deionized H_2O and add 1 ml HNO_3.

6.   Titrate with 0.1N $AgNO_3$ to the same voltage end point as determined from the standardization.

7.   Record the ml of $AgNO_3$ required for the titration.

Calculations

A.   For samples containing less than 5% NaCl, calculate % NaCl as follows:

$$\% \text{ NaCl} = \frac{(\text{g NaCl/ml AgNO}_3)(\text{ml AgNO}_3 \text{ for titration})(100)}{\text{g sample}}$$

where: g NaCl/ml $AgNO_3$ is from standardization

B.   For samples containing more than 5% NaCl, calculate % NaCl as follows:

$$\% \text{ NaCl} = \frac{(\text{g NaCl/ml AgNO}_3)(\text{ml AgNO}_3 \text{ for titration})(200)(100)}{(\text{g sample})(\text{ml aliquot titrated})}$$

Quality Control

A.   Record the weight of sodium chloride used for each standardiation run.

B.   Record the burette readings for each portion of $AgNO_3$ added for the first standardization.

C.   Record the mV readings corresponding to each added portion of $AgNO_3$ for the first standardization.

D.   Prepare a plot of ml silver nitrate against mV readings for one run.

E.   Determine the titration end point on the plot.

F.   Repeat the standardization at least twice more and calculate an average grams of sodium chloride that each ml of silver nitrate reacts with and record. For the second and third standardizations, just titrate to the end point determined from the first run.

G.   Recheck the standardization every three months and document.

Bibliography

Official Methods of Analysis (1984) 14th Ed., AOAC, Washington, D.C., secs. 7.106-7.108 Modified